EOARD Report
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DESCRIPTION OF THE RESEARCH

1.Introduction

There are many successes of nitrides in the field of light emitting diodes, blue lasers, high mobility transistors and UV detectors. The growth of nitrides by MOCVD, which has been intensively investigated in the last decade, contributed significantly to these outstanding successes. However, in spite of this progress, the fundamental reaction mechanism in the MOCVD growth of GaN and in particular the incorporation of Mg in p-type GaN are poorly understood.

The deposition of epitaxial GaN is traditionally carried out using mixture of trimethylgallium (TMG) and ammonia (NH_3). Hydrogen, besides nitrogen is predominantly used as the transport gas. Use of hydrogen as a transport gas and the pyrolysis of highly concentrated NH_3 results in a high concentration of molecular and atomic hydrogen near a substrate surface. Hydrogen at the surface of the growing film can influence the structural properties and the growth rate [1,2]. In addition to that, a growth temperature of about 1000C is higher than the decomposition temperature of metalorganic compound $Ga(CH_3)_3$ and their hydrocarbon ligands. This leads not only to desired bond breaking between the gallium atom and the methyl groups of the precursors, but also to pyrolysis of hydrocarbonds with incorporation of hydrogen and carbon into the GaN film [1]. Therefore, such complexes as CH, CH_2 and CH_3 may be incorporated in GaN layer. The role of such defect complexes in GaN is unknown.

Hydrogen as an impurity plays important role in many semiconductors. The ability of atomic hydrogen to passivate or compensate both shallow and deep defects and to induce extended defects has a large influence on optoelectronic properties of semiconductor materials [2]. It is known that in MOCVD – grown p type GaN the hydrogen plays a crucial role. The growth of p type GaN is based on doping with magnesium. In the MOCVD this is realized by using bis – cyclopentadienylmagnesim metalorganic. The GaN:Mg grown with help of this metalorganic is highly resistive which indicate that Mg acceptors are passivated. It has been demonstrated that the activation of Mg acceptors can be achieved by thermal annealing in the temperature range 600C – 750C [4]. Based on this observation it was established that hydrogen passivate the Mg acceptors. From theoretical investigation of Mg – H complexes it was concluded that hydrogen does not form a bond to the Mg atom directly, but prefers

antibonding site of one of the nitrogen neighbors [5].

This report describes electrical and photoconductive study of high resistive GaN:Mg. It describes also the investigation of localized vibrational modes in undoped, Si and Mg doped GaN layers.

2. Growth of GaN and GaN:Mg layers.

The growth of gallium nitride has been performed using modified Epigress atmospheric pressure MOCVD system. In the growth of GaN Al_2O_3 substrates are used.

The growth of GaN is performed in four steps.

The first step of the growth is connected with purification of the surface of Al_2O_3 substrate. The sapphire substrate is annealed in the H_2 flow at temperature close to 1100C for a few minutes.

After that, the temperature is lowered to 500C and a flow of ammonia and TMG is introduced. The low temperature GaN buffer layer of thickness close to 200A is grown. This part of the process completes the second step of the growth.

In the third step of the growth the temperature is increased to about 1050C (without flow of TMG) to anneal buffer layer for about 20 minutes in the flow of ammonia. In this stage of the growth the recrystalization of the buffer layer takes place.

The fourth step of the growth is connected with a proper growth of the layer. The temperature is kept close to 1050C and in addition to flow of ammonia the flow of TMG is introduced. The ratio of NH3 to TMG is kept close to 2000:1. The hydrogen is used as a carrier gas. The morphology of the surface was mirror like. From previous investigations we know that our layers have gallium polarity [6].

The thickness of the grown GaN layers is ranging between 1 – 3μm. For n type doping Si impurity from silane (SiH_4) and for p type doping Mg impurity has been used.

3. Properties of GaN:Mg layers.

As grown GaN layers heavily doped with Mg ($N=10^{19}$ cm^{-3}) have a very high resistivity due to passivation of Mg acceptors by hydrogen. This high resistivity material was used to investigate the photoconductive properties in the UV region. Metal Au/Ni contacts have been evaporated on the surface of the grown layer. The typical value of dark current in our detectors (under bias of a few volts) was close to 1 nA at room temperature. Contacts have not been alloyed because alloying always lead to partial activation of acceptors in the layer of GaN:Mg and to decrease of the resistivity the layer. That resulted in an increase dark current to an unacceptable high level.

Measurements of thermal activation of conductivity are shown in Fig. 1. During the first measurement of an as grown sample the current was activated with energy 1.81 eV, close to the half of the energy gap of GaN. Thus, one may conclude that the investigated material is intrinsic, but the measured current was significantly larger than that expected for the intrinsic material. Moreover, during cooling of the sample, it was observed that the current did not follow results obtained during heating, but was significantly larger. In the second measurement the conductivity was activated with energy 0.38 eV. Results obtained in subsequent measurements are similar to that obtained in the second measurement. That means that during the first measurement, some of Mg acceptors were activated. This result indicates that certain Mg – H complexes may dissociate at temperature close to 300C which is much lower than usually reported for activation of p-type conductivity in GaN:Mg. This new finding is crucial in understanding transport properties of high resistivity GaN:Mg.
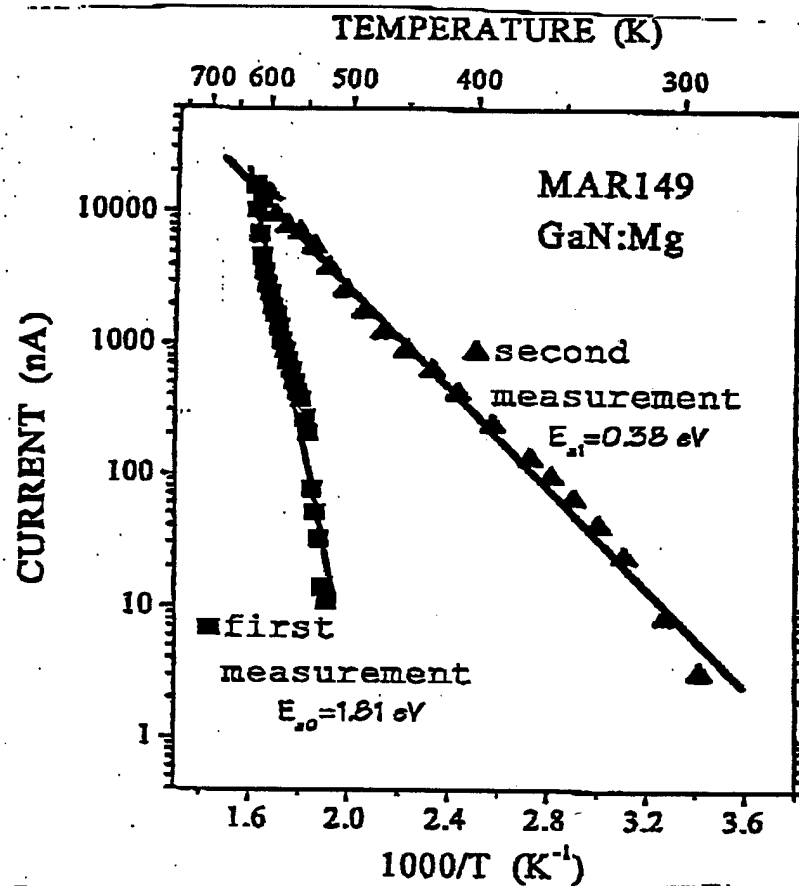
Fig. 1. Thermal activation of current in high
resistivity GaN:Mg.

The spectral sensitivity of samples has been investigated in the UV region and the representative one is shown in Fig 2. The intensity of incident light on the photodetector was measured with an UV – enhanced Si photodiode. The sensitivity monotonically increases as the wavelength is decreased down to 300 nm.
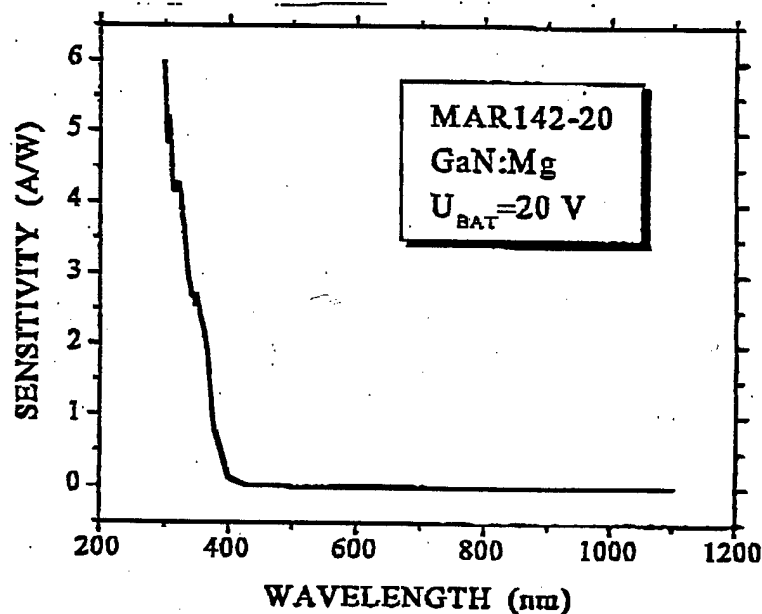

Fig. 2. Spectral dependence of sensitivity of
GaN:Mg photodetector.

A typically observed maximum at a wavelength corresponding to the energy gap is not observed in our detectors. The detector has photoconductive gain not higher than 10, which is a low value. The detector has only the virtue of being selectively sensitive in the UV region. However, the most serious problem of the photodetector is connected with a large time constant of the order of several minutes.

The estimated radiative recombination constant in GaN is equal to 10^{-8} cm^3/s [7]. Therefore, even for a very low concentration of free carriers of 10^{11} cm^{-3} the lifetime of free carriers should be equal to 1 msec, which is almost 6 orders of magnitude shorter than the observed lifetime in investigated GaN:Mg. Therefore, in order to explain the contradiction between the observed long lifetime and the expected radiative lifetime one has to postulate the presence of strong fluctuation of potential in unannealed GaN:Mg, which is schematically shown in Fig.3. The presence of fluctuations results in spatial separation of electrons and holes, which in turn reduces the recombination rate by many order of magnitude. The recombination takes place only in small regions of overlap between electrons and holes. Thermal excitation of carriers to the regions of presence of carriers of opposite sign leads to increase of recombination rate. Indeed, a significant decrease of the response time was observed, when the detector was heated up to 150C.
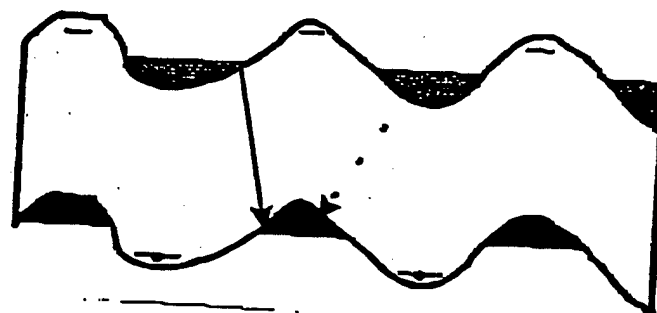

Fig. 3. Potential fluctuations in unannealed
GaN doped with Mg.

In order to shorten the time response of the GaN:Mg photodetector, the magnitude of potential fluctuation must be reduced. The microscopic origin of the fluctuation is not known and one may postulate the following possibilities:

1. One possibility is that the fluctuations are connected with fluctuation of Mg impurity in the GaN:Mg layer. In this case lowering the Mg doping level may reduce the fluctuation.
2. The second possibility is that fluctuations are connected with other than Mg impurities. In this case the localized vibrational mode (LVM) spectroscopy may be apply to identify defect complexes. This technique was used to characterize our GaN layers and results are descibed below.
3. The third possibility is that fluctuations are present in the buffer layer. Photocarriers may diffuse to the buffer layer and be trapped by fluctuations connected with dislocations and grain boundaries in the conductive buffer layer. In such case electrical insulation of the active GaN:Mg layer from the buffer layer may be required.

4.Local Vibrational Mode Spectroscopy

The LVMs spectroscopy has not been widely employed in studying dopants incorporation and impurities in GaN. The major problem associated with the study of the LVMs in GaN is that the substrate used to grow GaN is sapphire, which has a very strong infrared absorption band in the spectral region of $450 - 1500$ cm^{-1}. This band obscured many LVMs of simple substitutional impurities. However, hydrogen related defect complexes usually possess LVMs at wave numbers higher than 1500 cm^{-1}. Hence, it is possible to study impurity incorporation in GaN by monitoring the dopant – H complexes related LVMs. Hydrogen can be incorporated into III-V nitrides during the growth of the material.

The localized vibrational modes were measured at 77K using a BOMEM Fourier – transform infrared spectrometer in Air Force Research Laboratory Kirtland AFB. Samples which have been tested were undoped, high resistive GaN:Mg and low resistive n-type GaN:Si as well.

The three spectra shown in Fig. 4 were measured for undoped, GaN:Mg+H grown using hydrogen as carrier gas and GaN:Mg+O using nitrogen as carrier gas. Three distinctive peaks are observed at 2851.7, 2922.9, and 2956.5 cm^{-1}. The intensity of three peaks is sample dependent.
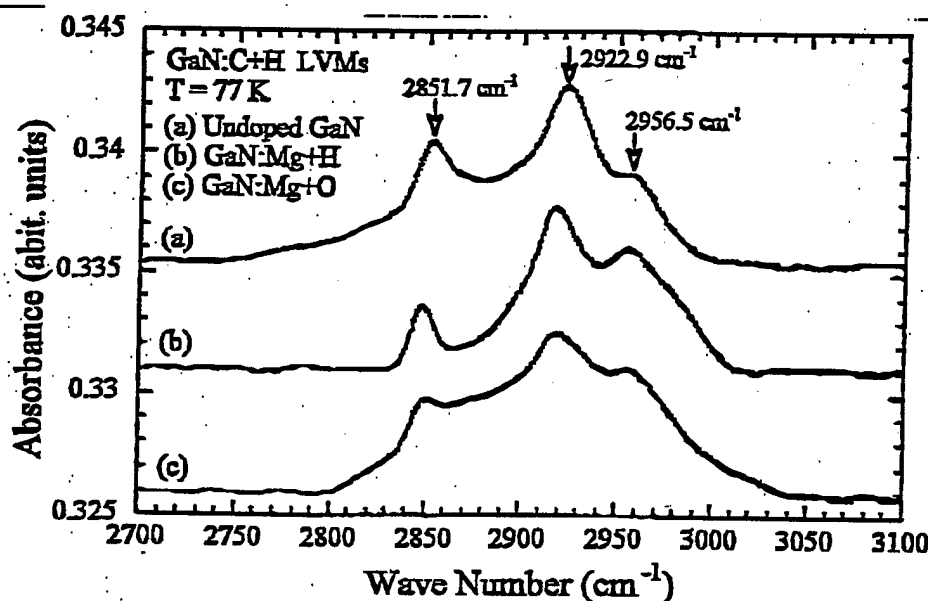

Fig. 4. Carbon – hydrogen localized vibrational mode spectra
measured at 77K in various samples of GaN

Such peaks were observed previously and tentatively assigned to the CH stretching mode, CH_2 asymmetric stretching mode, and CH_3 asymmetric stretching mode, respectively [8]. The same three peaks were found in low resistivity GaN:Si as well. The localized vibrational mode spectra related to C-H complexes are present in all investigated samples and are not connected with Mg doping. Therefore, potential fluctuation present in GaN:Mg are not caused by fluctuation of CH complexes. The shape and the peak position of these LVMs were found to be sample dependent. The sample dependence was explained in terms of local strains generated from high dislocation densities inherited in GaN samples. Localized

vibrational modes associated with Si-H in GaN:Si and Mg-H complexes in GaN:Mg have not been observed in the current study. The details of the LVMs investigations are described in our publication [9].

5.Summary

It has been found that the main obstacle on the way of obtaining good GaN:Mg UV photoconductive detector is a long time constant of the detector. The most likely strong potential fluctuations in high resistive GaN:Mg are responsible for the long lifetime of carriers.

One of important results of our research is finding that annealing of GaN:Mg already takes place at temperature close to 300C. That indicates that dissociation of some $H-Mg$ complexes take place at much lower temperature than it was up to now reported.

Finally, our report describes measurements of LVMs of undoped, Mg and Si doped GaN. It has been shown that CH complexes exist in MOCVD grown GaN layers irrespectively from doping.

6.Direction of future research

To improve performance of UV photoconductive detectors one has to reduce potential fluctuation present in the GaN:Mg layer. Two direction of future research will be made:
1. Reduction of Mg doing level to check if the lower concentration of Mg shorten the time constant of the detector.
2. Elimination of possible electrical transport through highly defective buffer layer on the sapphire/GaN interface. That can be achieved by growing a second AlN buffer layer (after the growth of about 1 μm of GaN:Mg). The second AlN buffer layer will isolate a layer of GaN:Mg grown on top of it from the strongly defected layer of GaN grown close to sapphire – GaN interface.

Search for localized vibrational modes due to Mg-H complexes in such higher quality GaN:Mg layer may be made.

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Localized vibrational modes of carbon-hydrogen complexes in GaN

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Localized vibrational modes of carbon-hydrogen complexes in metalorganic chemical vapor deposition grown GaN on sapphire were studied using a Fourier-transform infrared spectroscopy technique. Three distinctive localized vibrational modes were observed around 2850, 2922, and 2959 cm^{-1} for undoped, Si-, and Mg-doped samples. These peaks are related to CH, CH_2, and CH_3 defect complexes, respectively. However, the localized vibrational modes were not observed in some undoped samples, which is indicative of high quality grown epitaxial layers. It is also observed that the frequencies and intensities of the localized vibrational modes are sample dependent. © 1999 American Institute of Physics. [S0003-6951(99)00831-1]

The development of III–V nitride semiconductors and the emerging advances in a broad range of nitride-based electronic and optoelectronic devices have witnessed dramatic successes in recent years. One of the most important aspects of recent investigations of III–V nitrides is the ability to produce and fairly control n- and p-type doped materials. Massive research efforts on devices have been launched after Amano et al.[1] observed cathodoluminescence results from Mg-doped GaN. However, the dopant incorporation remains the subject of various investigations. The localized vibrational modes (LVMs) spectroscopy is a very powerful tool in identifying impurities and dopant incorporation in semiconductors. This is due to the fact that the LVMs spectroscopy is very sensitive to the atomic structure surrounding a dopant atom, the dopant location, dislocation density, and strain. As a typical example, the high-resolution LVMs spectrum of a substitutional acceptor carbon in GaAs exhibits a fine structure due to the Ga isotopic effect.[2]

The LVMs spectroscopy has not been employed aggressively in studying dopants incorporation and impurities in GaN and related compounds. One of the major problems associated with the study of the LVMs in GaN is that the substrate used to grow GaN is sapphire, which has a very strong infrared absorption band in the spectral region of 450–1500 cm^{-1}. This band obscured many LVMs of simple substitutional impurities and dopants such as Mg, C, and Si. Hydrogen related defect complexes usually possess LVMs at frequencies higher than 1500 cm^{-1}. Hence, it is possible to study dopants incorporation in GaN by monitoring the dopant-H complexes related LVMs. As an example, hydrogen can passivate Mg, which is an important acceptor in GaN. Hydrogen can be easily incorporated into III–V nitrides during or after the growth of the materials.[3] Additionally, atomic hydrogen can easily diffuse into the III–V nitrides during device processing, since most of the chemicals

that come into contact with the surface contain hydrogen.

In this letter, we report on the LVMs of carbon-hydrogen complexes in doped and undoped metalorganic chemical vapor deposition (MOCVD) GaN grown on sapphire substrate. The C–H LVMs were observed in samples doped with Mg, Si, or contaminated with O_2 and in undoped samples. However, these LVMs were not observed in undoped high quality samples as judged by the photoluminescence spectra. This suggests that the carbon is incorporated during the growth in the relatively lower quality samples. It is also observed that the frequencies and the intensities of the C–H LVMs are sample dependent, perhaps due to the variation of the dislocation densities and strains in the samples.

All the GaN samples tested in the present investigations were grown on sapphire substrates by the MOCVD technique at 1050 °C. The thickness of the GaN thin films ranged from 1 to 2.5 μm. The localized vibrational modes were measured at 77 K using a BOMEM Fourier-transform infrared spectrometer in conjunction with a continuous flow cryostat. Many samples were tested, but nine samples were selected for this study. Some of their characteristics are described in Table I. A special sample holder was constructed so that the incident light from the spectrometer

TABLE I. Characteristics of the samples used in the present study. The carrier concentration and mobility were measured at 300 K. The undoped samples are n-type with a typical concentration of $(1-8) \times 10^{17}$ cm^{-3}.

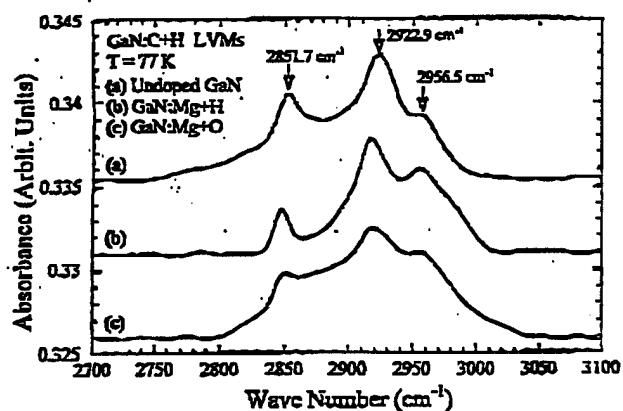| Sample	Dopant	Contaminant	Carrier gas	Carrier concentration $(\times 10^{17}$ $cm^{-3})$	Mobility (μ) $(cm^2/V s)$
A	undoped		H_2	7.0	53.0
B	Mg		H_2	3.4	24.0
D	Mg	O_2	H_2	12.0	
E	undoped	O_2	N_2		
F	undoped		H_2		
G	Si		H_2	12.3	350
J	Si		H_2	6.0	375
K	undoped		H_2	3.0	400.0
L	undoped		H_2	0.3	150.0

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FIG. 1. Carbon-hydrogen localized vibrational mode spectra of GaN samples A, B, and D measured at 77 K. Spectra (a), (b), and (c) were obtained for undoped sample (A), Mg-doped sample (B), and Mg-doped and O_2 contaminated sample (D), respectively. Hydrogen was used as a carrier gas during growth for the three samples.

reached the sample at the Brewster's angle. This configuration proved to be very useful to avoid the interference patterns generated from the substrate as well as from the GaN epitaxial thin layers. The LVMs were obscured by the interference patterns in spectra collected at the normal incident configuration.

The three spectra shown in Fig. 1 were measured for samples A, B, and D (see Table I). It is clear that three distinctive peaks are observed at 2851.7, 2922.9, and 2956.5 cm^{-1}. The intensity of the three peaks are sample dependent. The shapes of the spectra indicate that there are contributions from other LVMs, especially around 2900 cm^{-1}, in good agreement with previously published results[4] for C–H LVMs in vapor phase epitaxial GaN:Mg. Additional C–H LVMs spectra are plotted in Fig. 2. The spectra in Fig. 2 were obtained for samples E, F, and G, which are contaminated with O_2 [spectrum (a)], undoped [spectrum (b)], and Si-doped GaN [spectrum (c)], respectively. Figure 3 shows a spectrum obtained for a Si-doped GaN sample J. The shape of the spectrum in this figure is somewhat different than the spectrum observed for the Si-doped sample (G) in Fig. 2 [see spectrum (c)]

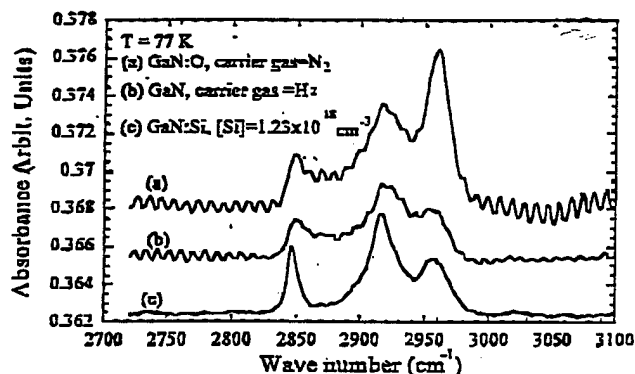Many of the doped and undoped samples obtained from

FIG. 2. Carbon-hydrogen localized vibrational mode spectra of GaN samples E, F, and G measured at 77 K. Spectra (a), (b), and (c) were obtained for undoped but O_2 contaminated sample (F), undoped sample (E),
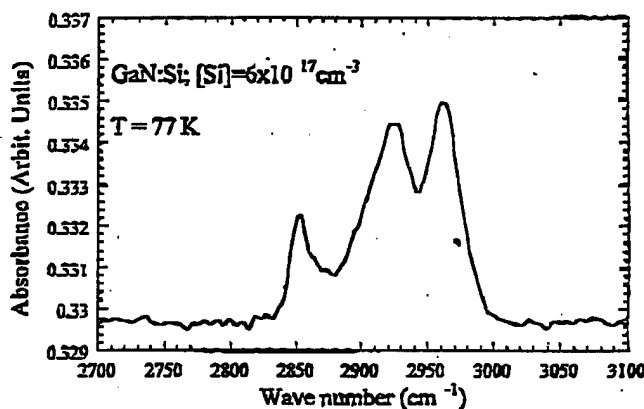

FIG. 3. Localized vibrational mode spectrum of CH complexes in Si-doped sample (J) measured at 77 K. The shape and the intensity of the peaks are different than the results reported in Figs. 1 and 2.

different sources were tested and found to contain the C–H LVMs. However, there were a few samples that do not show these LVMs. An example is shown Fig. 4, where two spectra of undoped samples are recorded at the normal incident configuration. The spectra are sloped due to the interference from the thin layers. The interference from the substrate in sample (K) [spectrum (a)] was not observed because the substrate backside was not polished. On the other hand, the interference from the substrate in sample (L) [spectrum (b)] is not large enough to cause any problems in the spectral region shown in the figure. This may be due to the fact that the substrate was polished at an angle (slightly wedged). It was observed that samples which do not exhibit C–H LVMs (see Fig. 4) possess the characteristic of narrower photoluminescence peaks near the band edge (free or neutral donor bound excitons' peaks) as compared to those of samples with C–H LVMs.

By careful inspection of Figs. 1–4, we observed that the intensity and the shape of the three dominant C–H LVMs in GaN are independent of the dopants, contaminants, and carrier gas. They are observed in both doped and undoped samples. However, undoped samples with narrow photoluminescence peaks near the band edge do not show the C–H LVMs. The full width at half maximum of the three peaks are also sample independent. Additionally, the intensities of the peaks are independent of the various dopants concentrations in agreement with Brandt et al.[5] For example, spectrum (c) in Fig. 2 is obtained for a Si-doped sample with [Si]
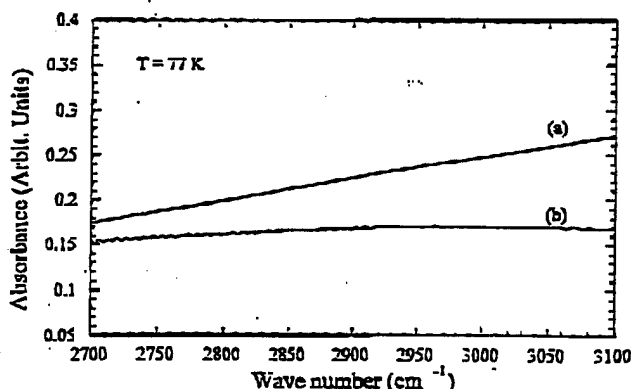

FIG. 4. Absorbance spectra of samples K [spectrum (a)] and L [spectrum

$=1.23\times10^{18}$ cm^{-3}, while the spectrum in Fig. 3 is obtained for a Si-doped samples with [Si]$=6.0\times10^{17}$ cm^{-3}. Yet, the intensities of the peaks in Fig. 3 are larger than those of spectrum (c) in Fig. 2. It is tempting to explain this behavior in terms of H trapping competition between C and Si substitutional atoms in GaN, but the results from other samples do not support this premise.

The three LVM peaks in the spectra in Fig. 1 observed at 2851.7, 2922.9, and 2956.5 cm^{-1} where tentatively assigned[4] to the CH stretching mode, CH$_2$ asymmetric stretching mode, and CH$_3$ asymmetric stretching mode, respectively. This assignment is based on a comparison between the LVM measurements in GaN and the calculated C–H frequencies[6] in α-Si$_{1-x}$C$_x$:H. However, we noted that the frequencies of the three peaks are sample dependent. It was found that the CH frequency could vary in different samples between 2847.0 and 2853.0 cm^{-1}, the CH$_2$ mode varies between 2910.0 and 2924.0 cm^{-1}, and the CH$_3$ mode varies between 2955.0 and 2963.0 cm^{-1}. The sample dependency of the LVM frequencies could be explained as follows. The typical dislocation densities in GaN are about 10^8–10^9 cm^{-3}. Hence, the atomic structures surrounding the C–H defect complexes are affected by the dislocations, which produce local strains. The strains associated with dislocations are usually sample dependent. These strains obviously affect the LVMs in a way that their frequencies are sample dependent.

It is believed that the H complexes in GaN are thermally more stable than in other semiconductor hosts.[7] The hydrogen and carbon in the present samples are incorporated during growth since the samples were not subject to any other processes. Hydrogen is well known to passivate dopants and impurities in semiconductors. For example, the hole compensation and the amount of incorporated H as a function of Mg concentration in GaN:Mg suggest the passivation of Mg by hydrogen.[8–10] However, the LVMs of Mg–H complexes in GaN have not been observed in the present GaN:Mg samples. On the other hand, Brandt *et al.*[5] reported two LVM peaks in the Raman and infrared spectra around 2168 and 2219 cm^{-1}, which were attributed to Mg–H LVMs. These frequencies, however, are not typical of Mg–H bonds.[7,11–13] They may be related to Si–H LVMs. But even with Si-doped samples (see Table I and Figs. 2 and 3), LVMs were not observed in the present investigation for either Mg–H or Si–H complexes.

With all samples (obtained from many sources) that were tested, only those with high quality photoluminescence spectra do not show the presence of C–H complexes related LVMs (see Fig. 4). We even tested the spectrometer for any possible contamination of hydrocarbons by running absorbance spectra without samples, with sapphire substrates, and with GaAs samples. It is clear, however, that the intensities

and the line shape of the LVMs of the C–H complexes in GaN are sample dependent. Additionally, carbon incorporation and the formation of C–H complexes have proven to be difficult for one to reach a concrete conclusion. Thus, further studies are needed to control doping and H passivation in GaN.

In conclusion, we presented localized vibrational mode spectra related to C–H complexes in MOCVD GaN. The C–H localized vibrational modes were observed in undoped, Si- and Mg-doped GaN. The shape and the peak positions of these LVMs were found to be sample dependent. The sample dependency was explained in terms of local strains generated from the high dislocation densities inherited in GaN samples. The C–H LVMs, however, were not observed in samples that exhibit photoluminescence spectra with narrow band edge peaks. Three distinctive LVM peaks observed around at 2851, 2922, and 2956 cm^{-1} where assigned to CH, CH$_2$, and CH$_3$ stretching modes, respectively. The CH$_2$ asymmetric frequency mode was found to vary the most in various doped and undoped samples. Localized vibrational modes associated with Si- and Mg-doped GaN were not observed in the current study. Additional LVM investigation is needed to understand the dopant incorporation and H passivation in III nitrides.

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13. ABSTRACT (Maximum 200 words)

This report results from a contract tasking University of Warsaw as follows: The contractor will investigate the growth of GaN material using atmospheric pressure metalorganic chemical vapor deposition method (MOCVD). The contractor shall develop and optimize Mg doped GaN epitaxial layers which will be able to serve as a base for sensitive photoconductive UV detector. The contractor will grow and perform basic characterization and photoluminescence of the GaN material. Samples will be delivered to Phillips Lab, at Albuquerque NM after the growth and electrical characterizations were made in the Institute of Experimental Physics at Warsaw, Poland.

14. SUBJECT TERMS

EOARD, Sensor Technology, Semiconductors, Optical Components, Optical Sensors

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